16 January 1986

Dr. Frank Baumann
California Department of Health Services
Southern California Laboratory
1449 West Temple Street
Los Angeles, CA 90026

Dear Frank:

Attached is a photocopy of the perchlorate analytical procedure used by CAL Analytical for the samples collected at the San Gabriel site.

For additional technical information on the method, please contact Tony Wong of CAL at (916) 372-1393.

Sincerely yours,

Original Signed by:

Laura J. Tom
Environmental Scientist
Quality Assurance Management Section
Environmental Services Branch
Office of Policy and Management

Attachment

cc: Dr. Ben Tamplin, Sanitation and Radiation Laboratory
Dr. David Spaeth

✓bc: Neil Ziemba (T-4-1)

Perchlorate Analysis

From 31 Dec 79 report to Acroject # 10954

Reagents:

a) 0.1% Crystal Violet (100 mg in 100 ml H_2O).
b) Phosphate buffer, pH 6.0; 34 g of KH_2 of PO_4 in 250 ml H_2O
c) Chlorobenzene
d) Stock perchlorate std, 1000 mg/L (prepared from $Mg(ClO_4)_2$)

Procedure:

1. Set up six 125 ml separatory funnels. To the first, add 50 ml of deionized water; this will be the blank. Prepare calibration standards from 0.01 - 0.10 ppm, as follows:

   Dilute 1.00 mL of stock to 1 liter in a volumetric flask. Place the following amounts into separatory funnels and add deionized water to give 50 mL ± 2 ml final volume:

   0.50 ml → 50 = 0.01
   1.0 ml → 50 = 0.02
   3.0 ml → 50 = 0.06
   4.0 ml → 50 = 0.08
   5.0 ml → 50 = 0.10

2. To each of the standards and the blank, add 2.0 ml phosphate buffer and 1.0 ml crystal violet reagent and mix well.

3. Add 5.0 ml chlorobenzene, stopper and shake for one minute. Do not open stopcock to release pressure. The funnel drain should be kept free of water. Allow the separatory funnels to stand for 15 minutes. Since the color in the chlorobenzene layer is quite stable, the solutions should be allowed to stand sufficiently long to give complete separation of the two phases

4. Drain 3 to 4 ml of the chlorobenzene layer into cuvettes and check closely for fine droplets of water. If found let the tubes sit for another 10 minutes before reading. Measure absorbance at 595 nm.

5. The separatory funnels can now be cleaned with deionized water and rinsed with acetone to remove adsorbed dye and drops of chlorobenzene. When dry, set up for sample analysis. To a clean separtory funnel add 10.0 ml of sample, or some dilution if the level is expected to be >0.1 ppm, and deionized water to give a final volume of 50 ml ± 2 ml. Repeat steps 2-4. Above procedure will allow a detection limit of 0.05 ppm perchlorate. Nitrate levels above 1.0 ppm will cause positive interference and must be corrected.